FACE-SHEET QUALITY ANALYSIS AND THERMO-PHYSICAL PROPERTY CHARACTERIZATION OF OOA AND AUTOCLAVE PANELS

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ABSTRACT

Increased application of polymer matrix composite (PMC) materials in large vehicle structures requires consideration of non-autoclave manufacturing technology. The NASA Composites for Exploration project, and its predecessor, Lightweight Spacecraft Structures and Materials project, were tasked with the development of materials and manufacturing processes for structures that will perform in a heavy-lift-launch vehicle environment. Both autoclave and out of autoclave processable materials were considered. Large PMC structures envisioned for such a vehicle included the payload shroud and the interstage connector. In this study, composite sandwich panels representing 1/16th segments of the barrel section of the Ares V rocket fairing were prepared as 1.8 m x 2.4 m sections of the 10 m diameter arc segment. IM7/977-3 was used as the face-sheet prepreg of the autoclave processed panels and T40-800B/5320-1 for the out of autoclave panels. The core was 49.7 kg/m^2 (3.1 lb/ft^3 (pcf)) aluminum honeycomb. Face-sheets were fabricated by automated tape laying 153 mm wide unidirectional tape. This work details analysis of the manufactured panels where face-sheet quality was characterized by optical microscopy, cured ply thickness measurements, acid digestion, and thermal analysis.

1. INTRODUCTION

PMC materials are increasingly utilized in large structures; driving the maturation of advanced material and processing technologies.[1] Efforts from both industry and U.S. Government teams have pushed Out of Autoclave (OoA) technology and application forward over the past several years; leading to manufacturing demonstrations including the Boeing Wing Spar[2] and the Advanced Composite Cargo Aircraft.[3]

Within NASA, composite structures for heavy-lift launch vehicles are projected to be the largest composite structures fabricated for aerospace. Specifically, the interstage of the Ares V Cargo Launch Vehicle was planned to be 10 m in diameter and 12 meters in height.[4] The size requirements of these structures have placed considerable emphasis on processing out of the autoclave. Manufacturing autoclave quality structures without autoclave pressure places added criticality to void removal. The generation of voids is dependent on a number of factors[5] including prepreg chemistry, tack, lay-up, and other considerations. This paper describes a manufacturing feasibility study of 1/16th arc segments of the 10 m Ares V interstage. Two material systems were selected; Cytec IM7/977-3 autoclave cure epoxy/ carbon fiber prepreg and Cytec T40-800B/5320-1 out of autoclave cure epoxy/ carbon fiber prepreg. This study details characterization of sandwich panels prepared from these materials and compares face-sheet consolidation in the autoclave and non-autoclave processed materials. Variation in tool-side and bag-side laminate quality is also noted. The data collected in this paper was used for structural analysis and modeling in preparation for larger, 1/6th segment, panel manufacture. Coupon test data on selected panels showed high strength values, indicative of good quality panel manufacturing in both the autoclave and out of autoclave systems.[6]

2. EXPERIMENTAL

2.1 Panel Fabrication

The composite tooling was built by Janicki Industries in Sedro-Woolley, WA, using a T300/Epoxy substructure with T300/Polyimide topcoat. Tool dimensions are approximately 2.5 m x 3 m with a curvature following a 5 m radius concave tool.

IM7/977-3 was selected for autoclave processing and was purchased from Cytec Engineered Materials as 145 gsm, 153 mm" wide, uni-tape prepreg. FM300M 0.08 psf film adhesive, was used to co-cure the aluminum core with the IM7/977-3 face-sheet.

T40-800B/5320-1 was also purchased from Cytec Engineered Materials as 145 gsm, 153 mm uni-tape prepreg and was used to evaluate the manufacturing feasibility of the panel section via a non-autoclave process. FM309-1 M 0.08 psf, film adhesive was used in the vacuum only panel fabrication.

Expanded, formed, and perforated aluminum honeycomb core (49.7 kg/m^3) was purchased from Alcore. A description of panel configuration and core thickness is given in Table 1.

Panels were fabricated at Hitco Carbon Composites, Inc, Gardena, CA, using an Automated Tape Laying (ATL) machine (Charger) built by MAG, as depicted in Figure 1. Six inch uni-tape was used to allow for fiber steering on the contoured tool.

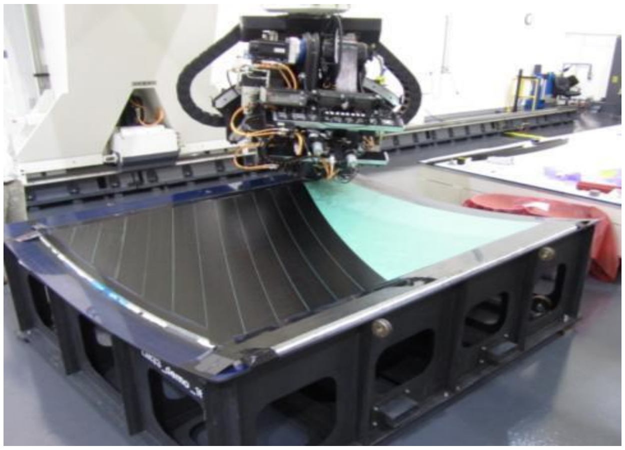


Figure 1: Automated manufacturing of contoured panel.

Table 1: Panel Nomenclature and Description

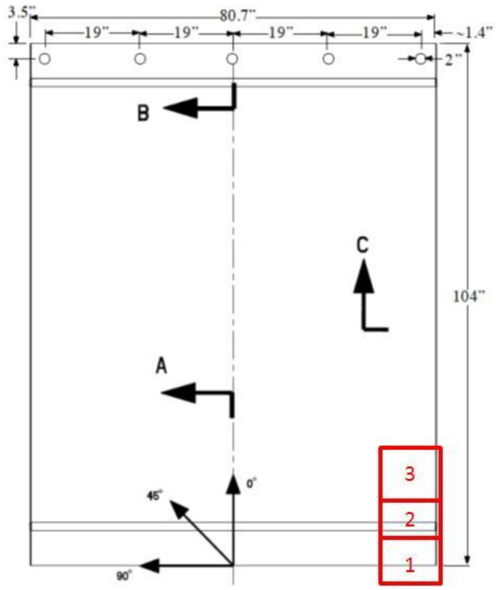
Panel	Prepreg	Core	Layup
8000CMDP	IM7/977-3	25.4 mm	$[45,90,-45,0]_s$
MTP-6001	IM7/977-3	41.3 mm	$[60,-60,0]_s$
MTP-6003	IM7/977-3	28.6 mm	$[60,-60,0]_s$
8010CMDP	T40-800/5320-1	25.4 mm	$[45,90,-45,0]_s$
MTP-6010	T40-800/5320-1	28.6 mm	$[60,-60,0]_s$
MTP-6000	IM7/977-3	28.6 mm	$[60,-60,0]_s$

Buildups on the ends of the 8 ply panels consisted of a balanced and symmetrical layup intertwined with the original 8 plies for a total of 16 plies. The buildups were approximately 178 mm wide and tapered into the acreage.

2.2 Face-sheet Characterization

2.2.1 Core and Adhesive Removal

Sections were removed from the edge of each manufactured panel for characterization of the composite skin, as shown in the schematic in Figure 2. The tool and bag sides were noted. The core was removed from each section and the face-sheets were sectioned for optical microscopy, acid digestion, and thermal analysis.



2.2.2 Optical Microscopy

Representative sections of the panel were polished for optical microscopy. Specimens were polished and photographed using an optical microscope. Thickness measurements were taken by a pixel counting method on both tool and bag side face-sheets. Five thickness measurements were taken at each location and averaged. Ply thicknesses were calculated by dividing the measured laminate thickness by the number of plies.

2.2.3 Void Analysis

The void volume and fiber content of each laminate panel was calculated following ASTM D 3171-76, Standard Test Method for Constituent Content of Composite Materials, with six samples tested per material. The matrix material was digested in hot sulfuric acid and hydrogen peroxide solutions and the remaining carbon fibers were filtered through a fine mesh screen. The fibers were flushed with water followed by an acetone rinse. The acetone was evaporated overnight in a fume hood and the fibers were then dried in an oven at 100 ^{o}C prior to weighing.

2.2.4 Dynamic Mechanical Analysis

DMA tests followed conditions specified in ASTM D7028-07, Standard Test Method for Glass Transition Temperature (DMA T_g) of Polymer Matrix Composites by Dynamic Mechanical Analysis. The test included a 5 ^{o}C/min ramp from room temperature to 50 ^{o}C above Tg. The frequency was set at 1 Hz, per the ASTM specification and the amplitude set at 20μm. Test specimens were dried at 70 ^{o}C prior to analysis. A single cantilever fixture was used for all DMA testing.

3. RESULTS AND DISCUSSION

3.1 IM7/977-3

Four panels were fabricated with IM7/977-3 prepreg and cured in an autoclave. A representative panel section image is shown in Figure 3. Considerable resin bleed-out was noted within the sections received for analysis; both along the core and soaked into the peel ply. Some resin flow is not unusual for panels made with this material, as the resin viscosity drops during the initial stages of the cure cycle.[7]

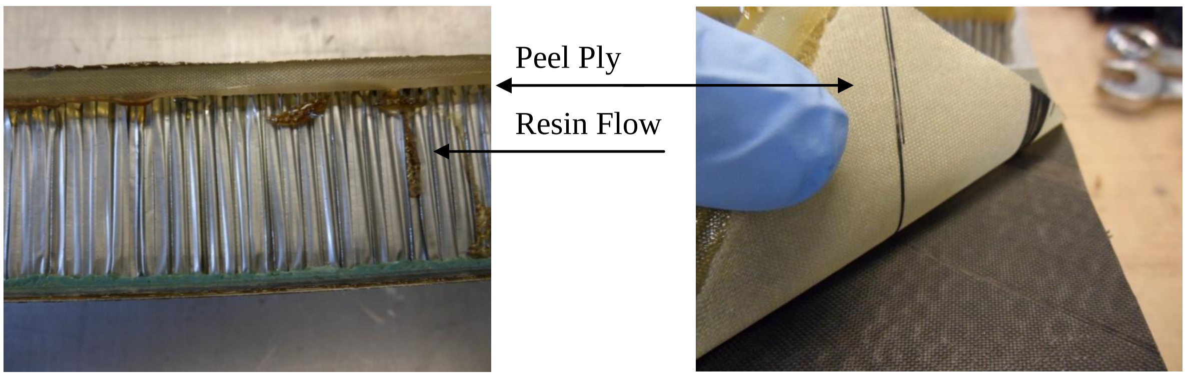
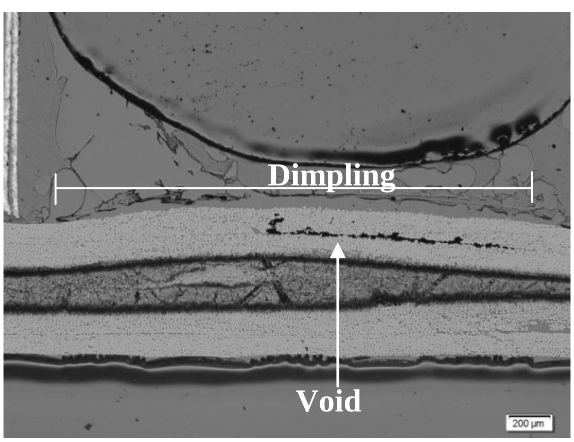
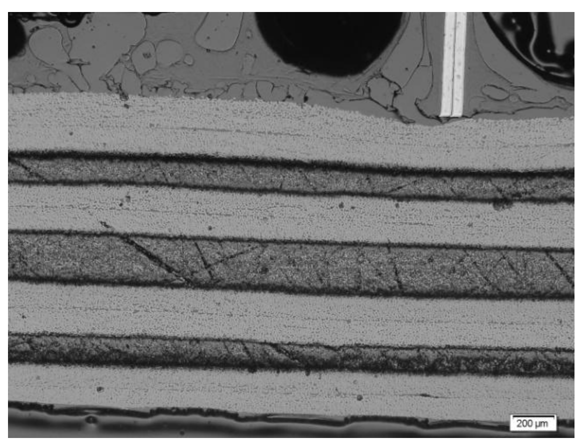


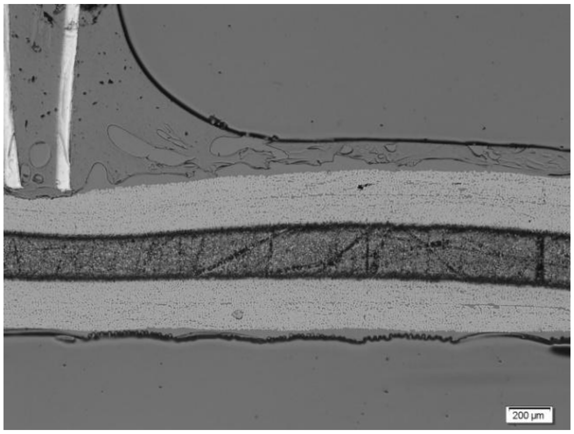
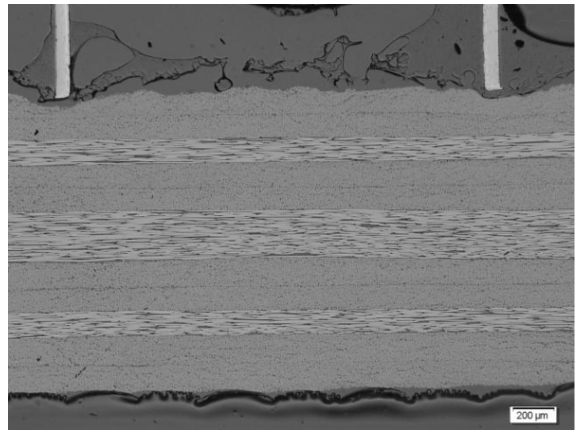
Figure 3: Resin flow along un-cut edge of IM7/977-3 panel.

3.1.1 Void Analysis

Optical microscopy images of IM7/977-3 face-sheet sections are shown in Figure 4, representing 6 ply and 12 ply regions of panel MTP6000. Voids are visible within the cross-sections, predominately between plies. Typical dimpling between nodes is called out in Figure 4a. There was not a notable difference in quality between the bag (Figures 4a and 4b) and tool side (Figures 4c and 4d) with respect to void content.

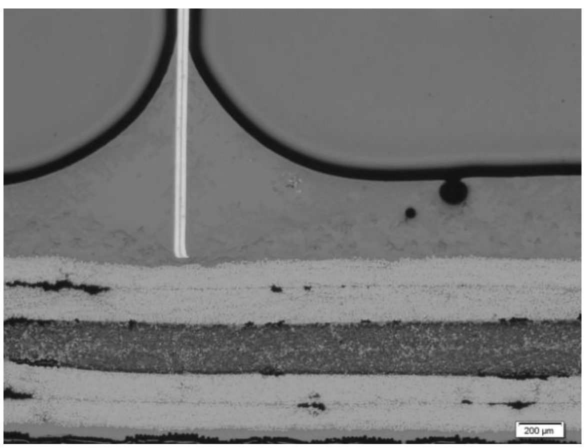
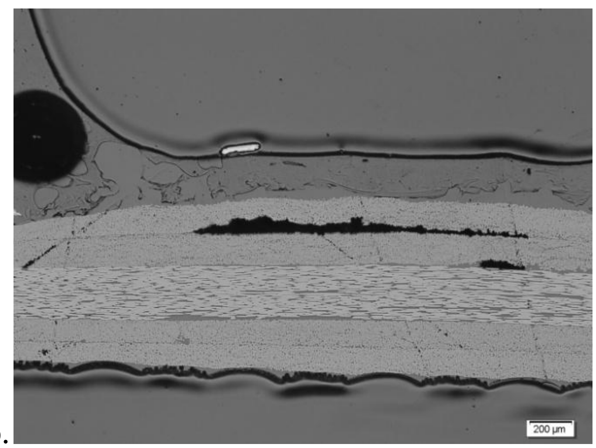
a. 

b. 

c.  d. 

Figures 4a – 4d: Photomicrographs representing bag (4a and 4b) and tool (4c and 4d) side sections of an IM7/977-3 panel.

Several images of the panel cross sections showed areas of face-sheet delamination and considerable void content, Figures 5a and 5b. However, IR thermography of the entire panel indicated no significant flaws; and the higher void content may be related to the edge section of the larger panel where these pieces were taken.

a.  b. 

Figures 5a – 5b: Photomicrographs show voids within an IM7/977-3 panel.

Acid digestion of these sections resulted in a measured void content below 2%, with no significant difference between the tool and bag side of the panel. Acid digestion results are shown in Table 2 for the IM7/977-3 panels.

Table 2: Acid digestion results of the IM7/977-3 skins.

Panel	Face-sheet Density (g/cm^3)	Average Void Content (%)	Average Fiber Vol. (%)	Average Resin Wt. (%)
8000CMDP_Bag	**1.59**	**1.8**	**65.5**	**27.0**
8000CMDP_Tool	**1.60**	**1.1**	**65.2**	**28.2**
MTP-6001_Bag	**1.61**	**1.0**	**64.5**	**28.5**
MTP-6001_Tool	**1.61**	**0.9**	**64.6**	**28.5**

MTP-6003_ Bag	**1.57**	**0.7**	**65.8**	**27.6**
MTP-6003_Tool	**1.58**	**0.9**	**65.6**	**27.6**
MTP-6000_Bag	**1.60**	**1.0**	**64.4**	**28.7**
MTP-6000_Tool	**1.60**	**0.9**	**64.2**	**28.8**

The acid digestion data calls out a higher fiber volume than the expected 60 vol.% and a lower resin weight content than anticipated.

The significant visible resin bleed-out shown in Figure 3, and the low resin content reported by acid digestion, prompted repeat measurements based on samples taken from within the interior of panel MTP-6000. The resin content of those sections by acid digestion was 28.5 wt. %; comparable to that at the edge. Average fiber volume of the interior samples was 64.0 vol.%.

3.1.2 Cured Ply Thickness (CPT)

The manufacturer supplied the average ply thickness as 0.131 mm (5.14 mil). Cured ply thickness (CPT) was measured by optical microscopy and was lower than anticipated; likely due to the resin bleed-out and measured low resin content.

Trends in the CPT measurements, Figure 6, were consistent between the four IM7/977-3 panels; with the bag-side (BS) skins approximately 2% thinner than the tool-side (TS). Overall, the average face-sheet thickness measured 4.5% thinner than the theoretical CPT. The range in skin CPT varied from 1% to7% lower than theoretical.

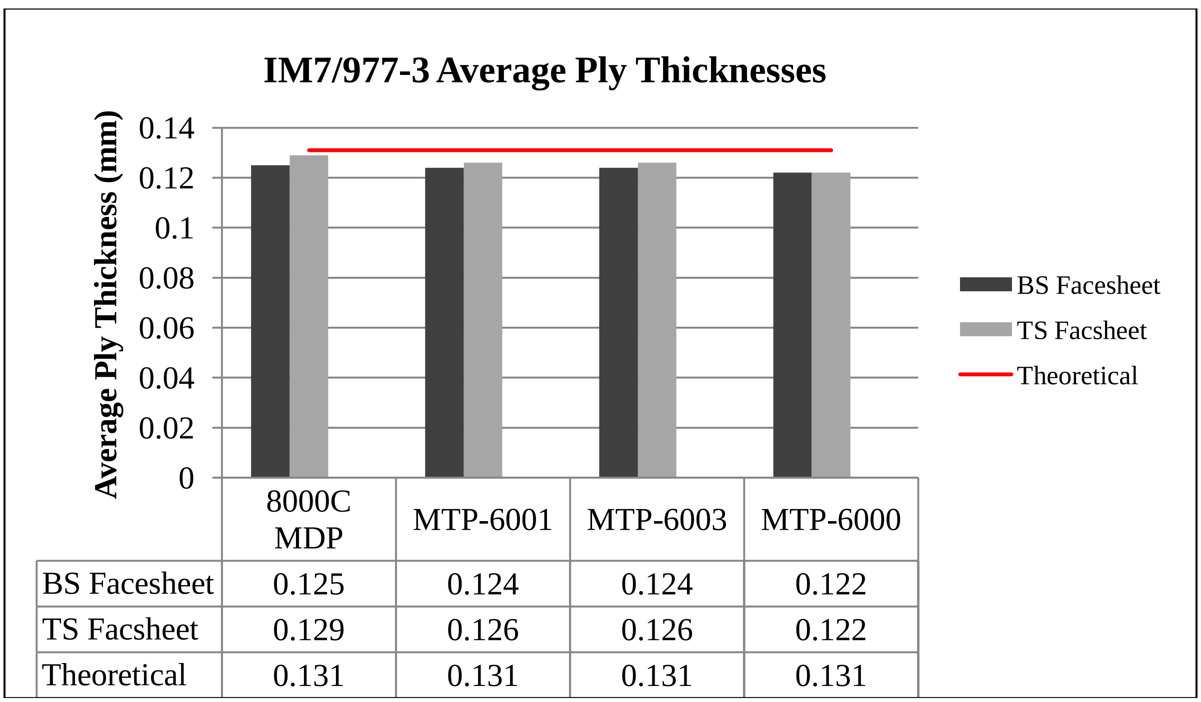


Figure 6: Average ply thickness for the IM7/977-3 panels.

3.1.3 DMA

The IM7/977-3 face-sheet Tg's are listed in Table 3. Tg was determined by the drop in storage modulus from a DMA curve.

Table 3: Tg as determined by DMA

Panel #	Tg (Bag-side) (°C)	Tg (Tool-side) (°C)
8000CMDP	166	179
MTP6001	173	173
MTP6003	175	172
MTP6000	195 (6 ply region) 192 (12 ply region)	196 (6 ply region) 207 (12 ply region)

The Tg Cytec lists for the IM7/977-3 neat resin is 178 °C. However, past work with this prepreg system has lead to a Tg just over 200 °C.[4] Representative DMA curves are shown in Figure 7.

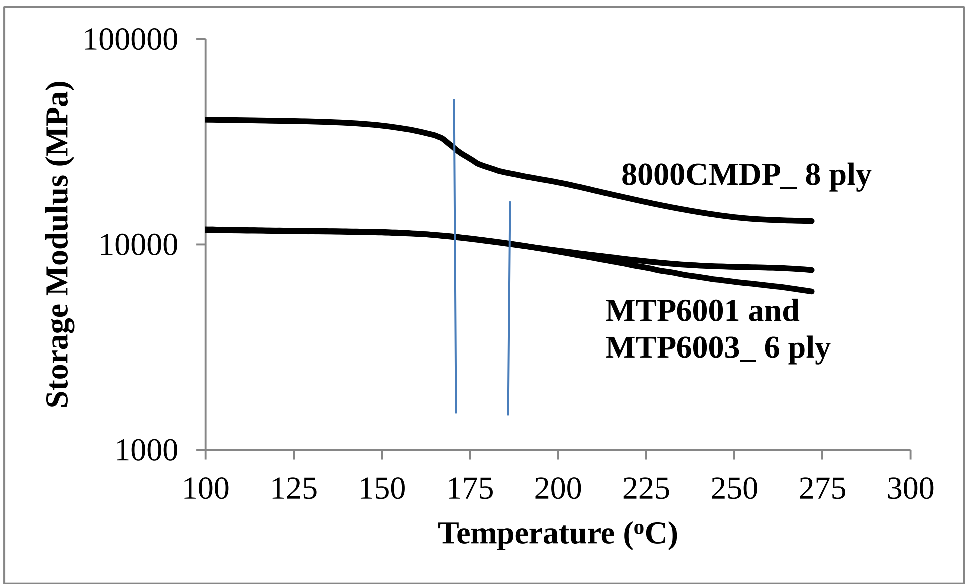


Figure 7: DMA curves of IM7/977-3 coupons

3.2 T40-800B/5320-1

Two panels were manufactured with T40-800B/5320-1 OoA cure prepreg tape. The fabricated panels did not exhibit the same level of resin bleed-out as was observed in the IM7/977-3 panels.

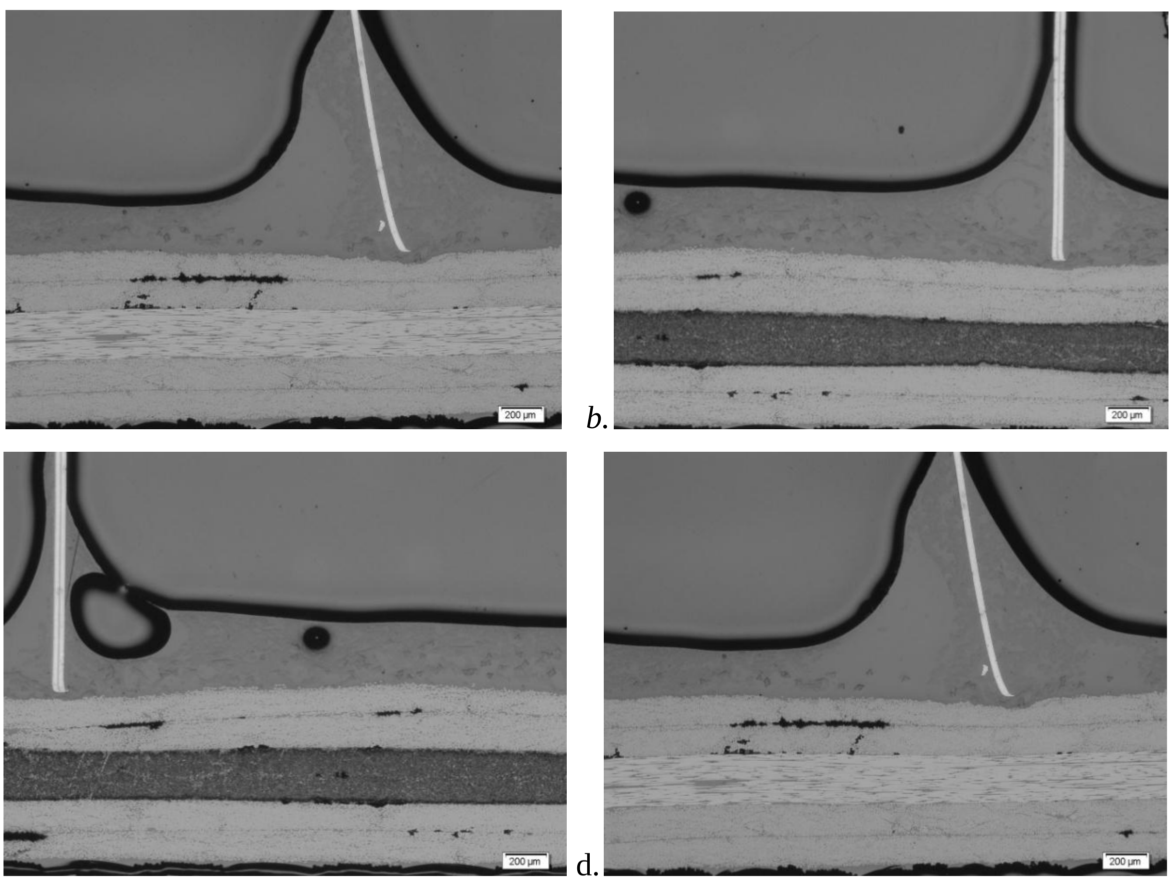
3.2.1 Void Analysis

The results of the out of autoclave panel were very similar to those of IM7/977-3. Optical microscopy images showed voids and delamination on both the tool side and bag side of the panel.

Average fiber volume, resin content, and void content by acid digestion are listed in Table 4. The void content was, in general, greater than that of the autoclave cured panels; however, void contents did not exceed 2%. The fiber volume and resin content in the T40-800B/5320-1 panels was representative of manufacturer specifications, likely due to the lower resin flow visible during cure. Optical photomicrographs are shown in Figures 8a – 8d.

Table 4: Acid digestion results of the T40-800B/5320-1 panel skins.

Panel	Face-sheet Density (g/cm^3)	Average Void Content (%)	Average Fiber Volume (%)	Average Resin Wt
8010CMDP_Bag	**1.61**	**1.0**	**62.0**	**31.2**
8010CMDP_Tool	**1.59**	**1.8**	**62.0**	**30.6**
MTP-6010_Bag	**1.60**	**1.8**	**60.7**	**31.6**
MTP-6010_Tool	**1.60**	**1.5**	**60.9**	**31.6**



a. *b.* c. d.

Figures 8a – 8d: Photomicrographs representing bag (8a and 8b) and tool (8c and 8d) side sections of and T40-800b/5320-1 panel.

3.2.2 Cured Ply Thickness

Theoretical cured ply thickness (CPT) of the OoA material was 0.136 mm (5.37 mil). Results of optical CPT measurements are given in Figure 9. As with the IM7/977-3 panels, there was a tendency for the bag-side skin to be thinner than the tool side skin. The MTP6010 panel (6 ply) face-sheet essentially matched the theoretical CPT. The 8 ply, 8010CMDP, was almost 10% below theoretical CPT on the bag-side, and 5% below CPT on the tool side. This variation may be due to the use of a rigid caul sheet in the bagging sequence for 8010CMDP.

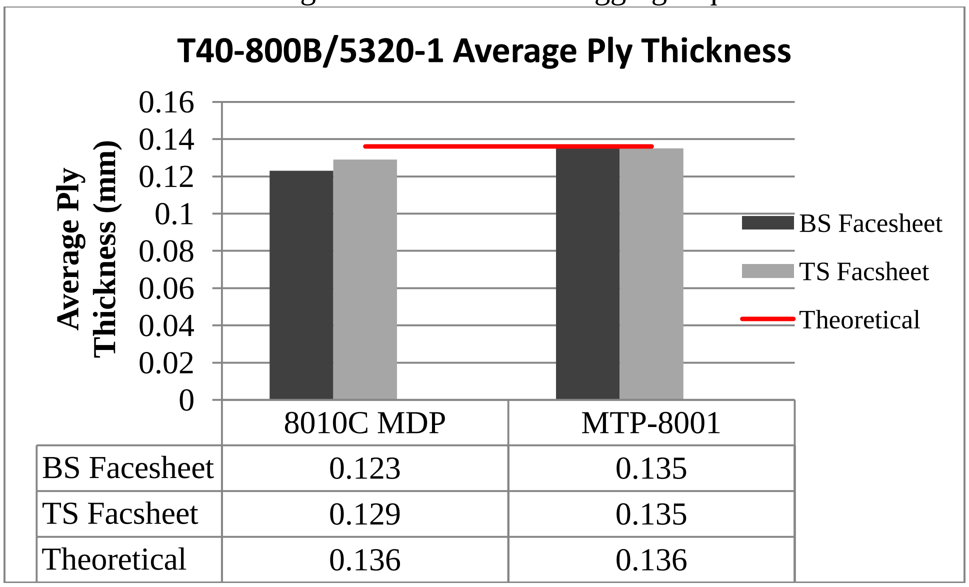


Figure 9: Average ply thickness for the T40-800B/977-3 panels

3.2.3 DMA

The T40-800B/5320-1 face-sheet Tg's are listed in Table 5. Tg was determined by the drop in storage modulus from a DMA curve.

Table 5: Tg for T40-800b panels

Panel #	Tg (Bag-side) (°C)	Tg (Tool-side) (°C)
8010CMDP	187	187
MTP6010	188	189

The Tg measured in this study was on the low end of what would be considered an acceptable Tg for this material.

CONCLUSIONS

As part of a larger manufacturing study, comparison of sandwich panel face-sheets, prepared using autoclave cured IM7/977-3 and vacuum bag only processed T40-800b/5320-1, were made and included void content, CPT measurement, and thermal analysis. In general, the measured CPT ranged from 3% - 5% below theoretical value. CPT below theoretical values was consistently observed in the IM7/977-3 panels and attributed to resin bleed-out during cure. This also led to a greater than anticipated fiber volume, and reduced resin content. This was observed in samples taken from both the panel edge and mid-section.

While some degree of porosity was observed on all panels, the vacuum only panel resulted in a generally higher void content. There was little difference noted throughout the analysis between the tool-side and bag-side skin quality.

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